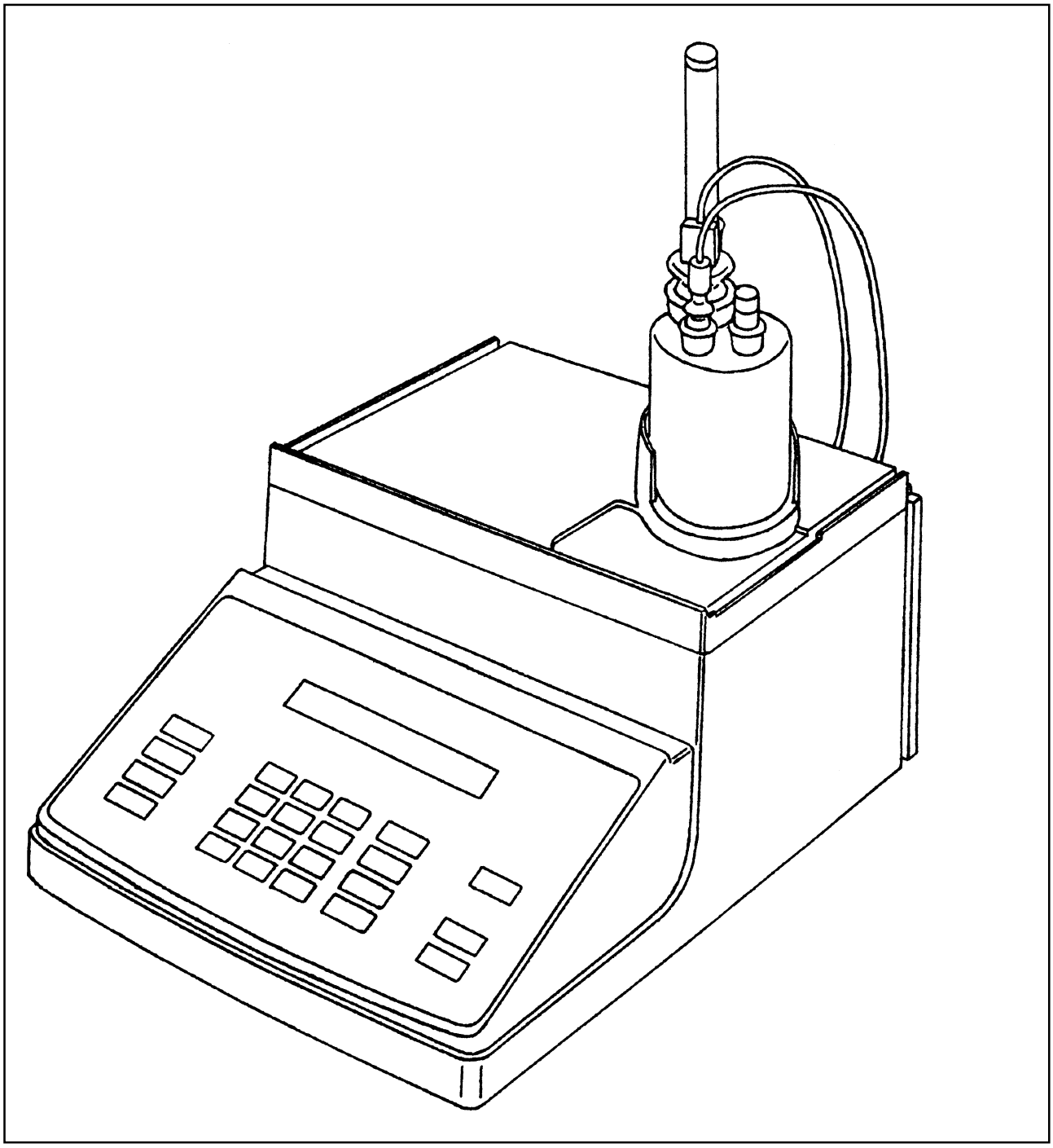
Operating Instructions

METTLER TOLEDO
DL36 KF Coulometer



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1. Introduction

Moisture in sugar or salt influences its pouring properties. Traces of moisture in brake fluid have an adverse effect on the functioning of vehicle brakes. Impurity water in the hydraulic oil of aircraft leads to a dangerous lowering in the performance of the hydraulic system. As the quality of products frequently depends on their moisture content, it is essential that this be known as accurately as possible. The DL36 KF Coulometer can be used to determine trace moisture with high accuracy.

The DL36

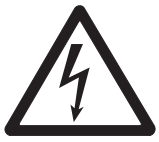
- can determine the moisture content of a sample in a range from 10 µg to 100 mg H_2O,
- allows entry of the sample weight before or after the titration either manually or automatically via an attached balance,
- automatically determines the drift and takes it into account in the result calculation,
- permits a recalculation of the results.

An attached printer prints out defined parameters and records the titration data in short-form or GLP format. An attached computer can interchange data with the titrator.

2. Safety measures

The DL36 has been tested for the experiments and intended purposes documented in the Operating Instructions. However, this does not absolve you from the responsibility of performing your own tests of the product supplied by us regarding its suitability for the methods and purposes you intend to use it for. You should therefore observe the following safety measures.

Measures for your protection



– Ensure that you plug the power cable supplied into a receptacle outlet that is grounded! In the absence of grounding, a technical fault could be lethal.
– Switch the instrument off and disconnect the power cable before you change blown fuses! An electric shock could be lethal.

– Never work in an environment subject to explosion hazards! The housing of the instrument is not gas tight (explosion hazard due to spark formation, corrosion caused by the ingress of gases).

– When using chemicals and solvents, comply with the instructions of the producer and the general lab safety rules!

Measures for operational safety

– Check the set operating voltage before you switch on the instrument! The instrument may suffer damage if the operating voltage does not match the line voltage.
– Use only fuses of the type specified in the Operating Instructions! (Risk of fire)
– Have the instrument serviced only by METTLER TOLEDO Service!
– Always wipe off splashed liquids immediately! The instrument is not water-proof.
– Exclude the following environmental influences:
  • powerful vibrations,
  • direct sunlight,
  • atmospheric humidity greater than 80%,
  • temperatures below 5 °C and above 35 °C,
  • powerful electric or magnetic fields!

3. Measurement principle

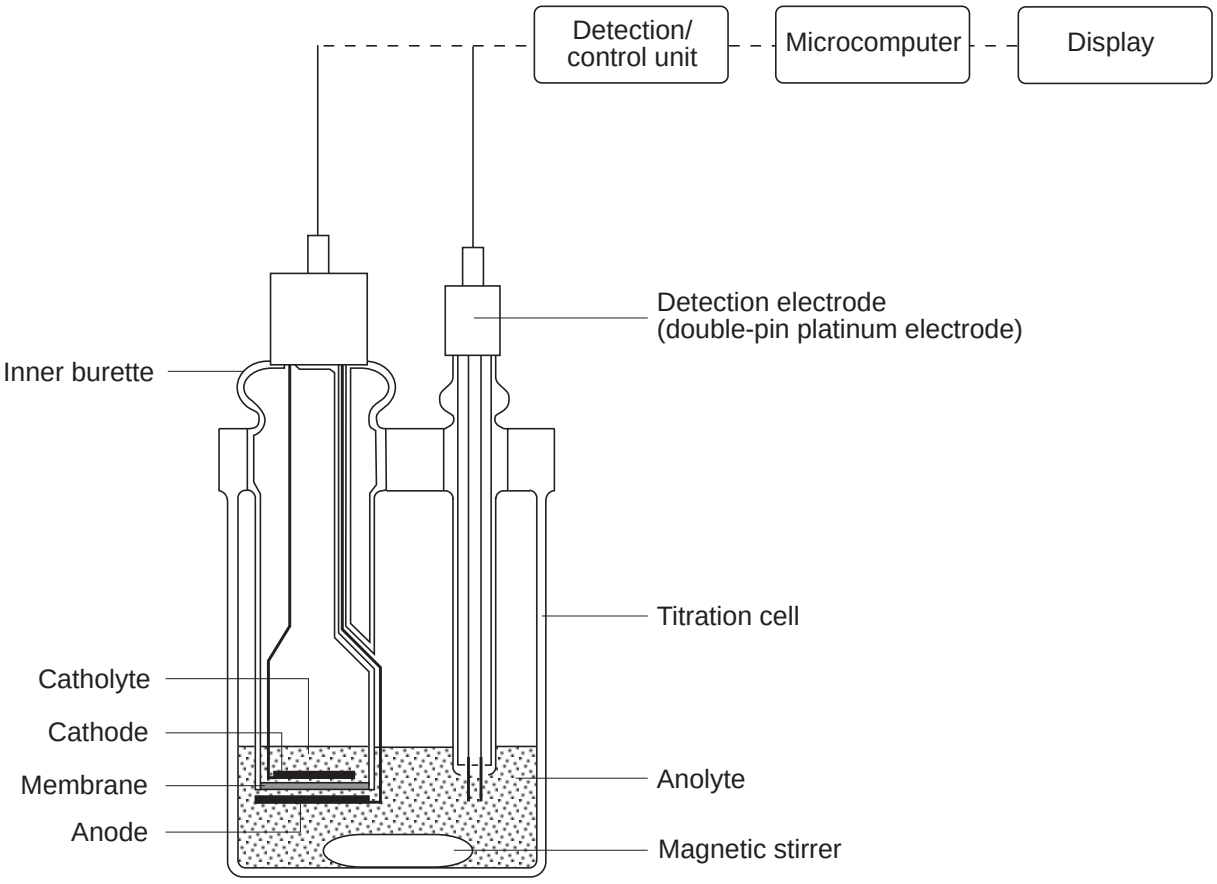
In the Karl Fischer method for determination of the moisture content, water (H_2O) reacts with iodine (I_2) and sulfur dioxide (SO_2) in the presence of methanol (CH_3OH) and a base (RN).

$$H_2O + I_2 + SO_2 + CH_3OH + 3RN \rightarrow [RNH]\,SO_4CH_3 + 2[RNH]I \qquad (1)$$

In the volumetric titration, iodine is added as the titrant. In the coulometric titration, iodine is generated electrolytically by an iodide-containing anolyte.

$$2I^- \rightarrow I_2 + 2e^- \qquad (2)$$

As long as water is present in the titration cell, the generated iodine reacts according to reaction (1). As soon as all the water has been consumed by the reaction, there is a small excess of iodine in the anolyte. The double-pin platinum electrode detects this iodine excess and the iodine generation is stopped. According to Faraday's law, the amount of iodine generated is proportional to the current which has flowed. In reaction (1), I_2 and H_2O react with each other in proportion 1:1. One mole of water (18 g) thus corresponds to 2 x 96 500 coulomb, in other words per mg H_2O a quantity of electricity of 10.72 coulomb is consumed. The total current consumption is a measure of the amount of moisture present.



4. Putting into operation

4.1 Preparing the titration cell

WARNING Never inhale the vapors of Karl Fischer reagents and avoid skin contact! The reagents could hazard your health.

– Remove the Teflon stopper of the titration cell and carefully slip the magnetic stirrer into the cell; replace the stopper.

– Place the titration cell in the holder.

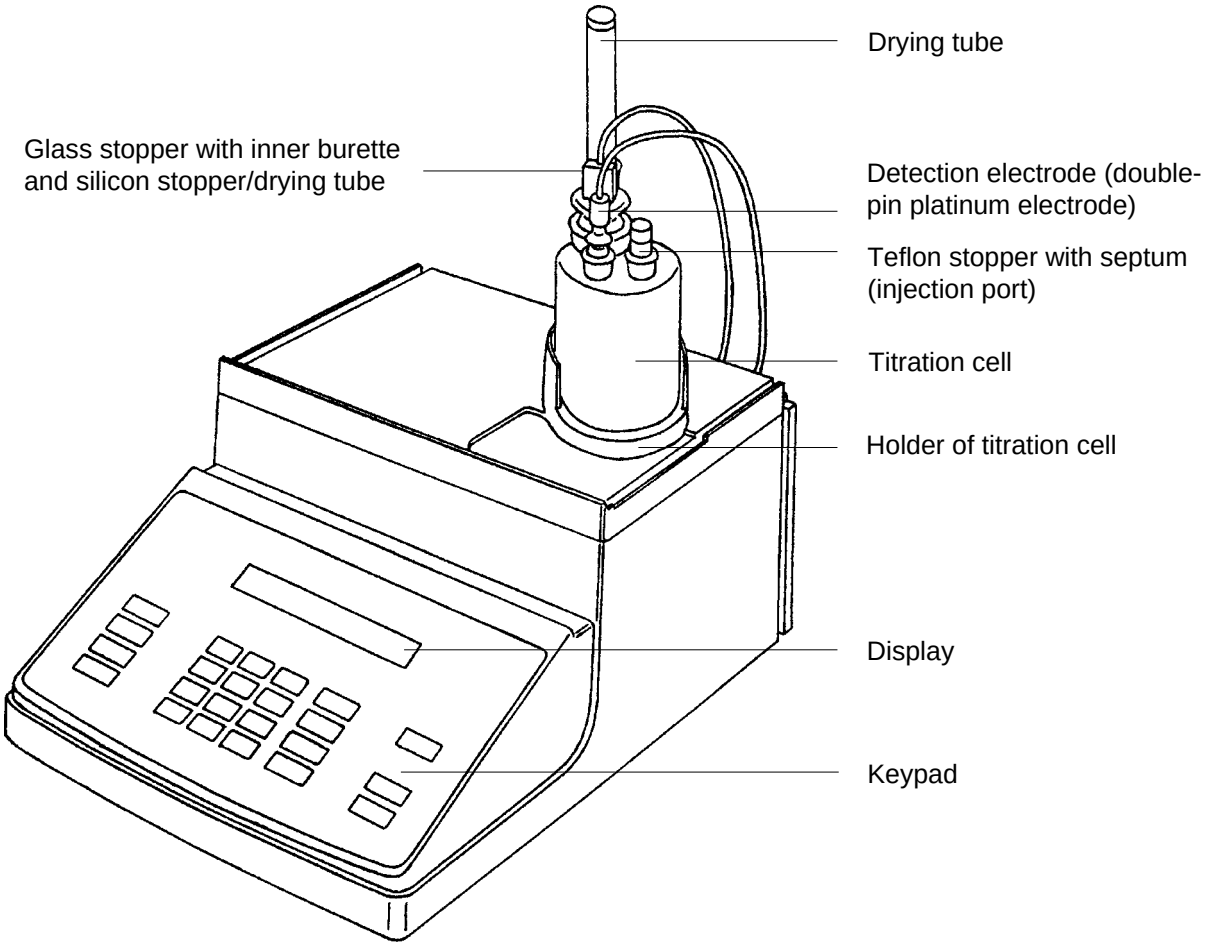
When the DL36 is delivered, the glass joints for the double-pin platinum electrode, inner burette and Teflon stopper have a very thin coating of silicon grease to assure tightness.

– Remove the Teflon stopper and add anolyte up to the lower mark on the titration cell (100 mL) using the funnel.

– Remove the silicon stopper from the inner burette and add 5 mL catholyte to the cathode of the inner burette; its level should be just **below** that of the anolyte.

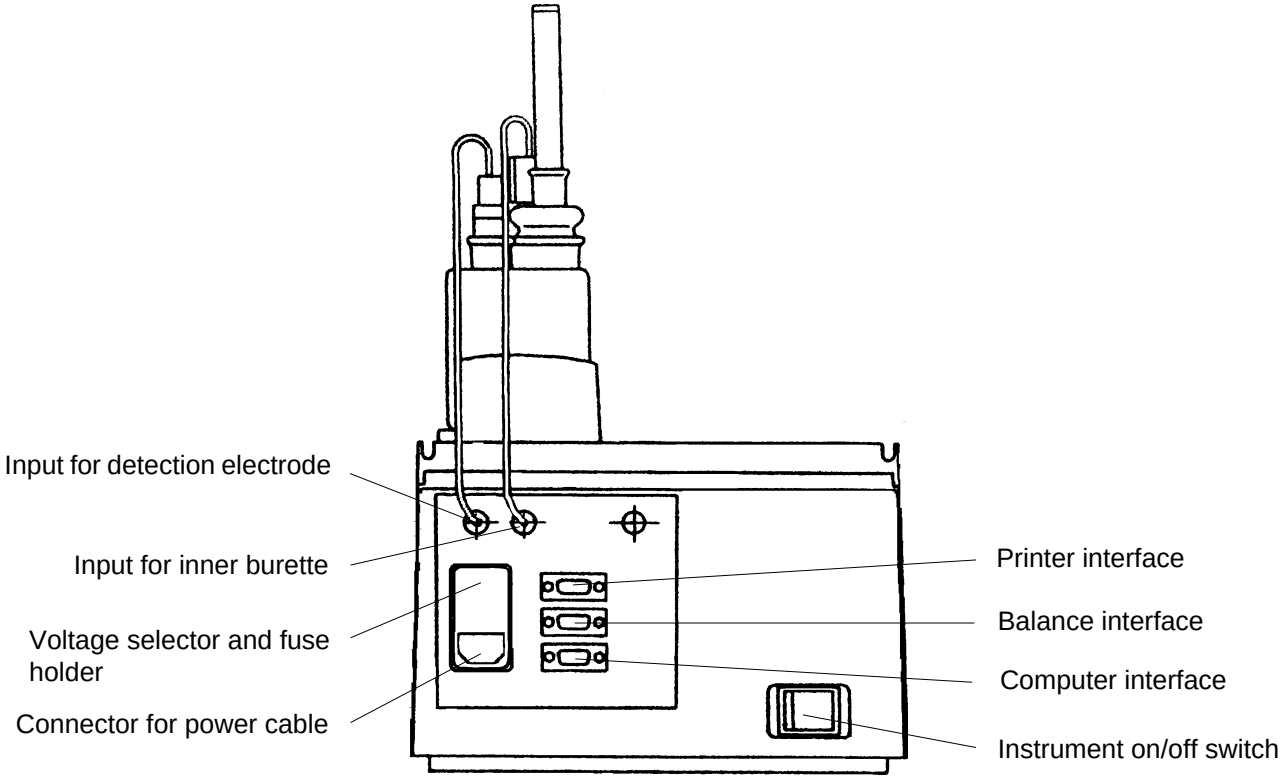
– Check that the glass joint is greased, then insert the drying tube.

– Plug the cables of the electrode and the inner burette into the appropriate inputs at the rear of the DL36 (see illustration on next page).



4.2 Attaching peripheral devices

You can attach, e.g. the GA42 Printer and an analytical balance from METTLER TOLEDO and a computer (see Section 8.5: *Selecting peripheral devices* and Section 12: *Standard and optional equipment*).



4.3 Switching on the instrument

The DL36 operates in a voltage range of 100 - 120 or 220 - 240 V.

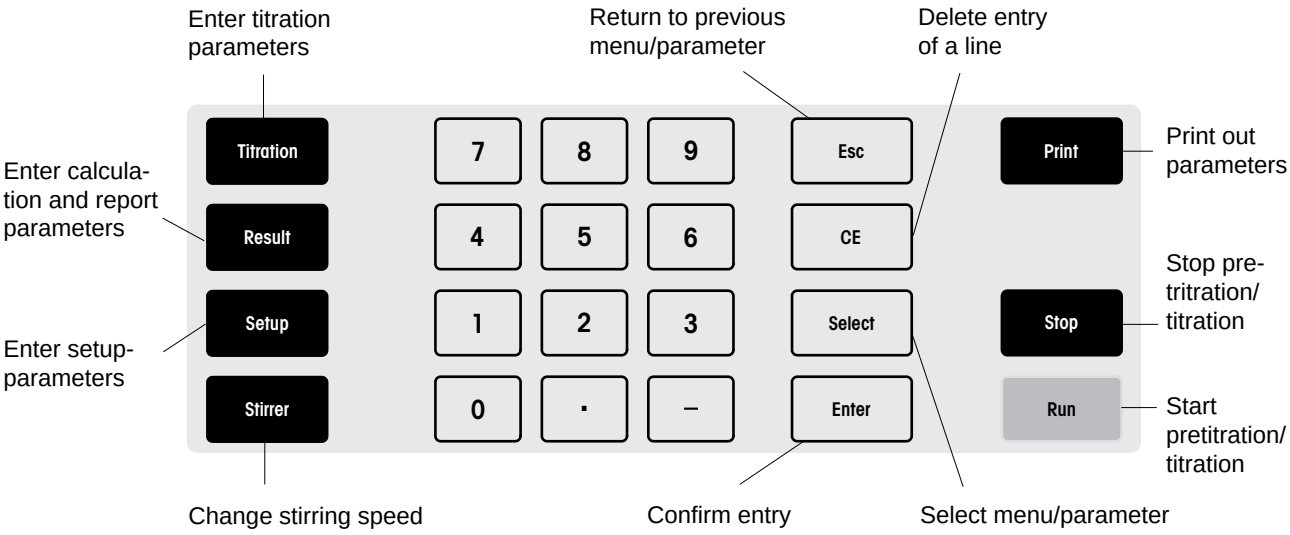
– Check the operating voltage set on the voltage selector before switching on the instrument! (See Section 11: *Maintenance and servicing.)*

– Plug in the power cable, connect to the power supply and switch on the instrument. The following appears in the display:

DL36 V1.0	followed by
< Run >	This display flashes until you press the **Run** key.

5. Keypad and display

5.1 The keypad



Stirrer

When you press this key, you can activate the stirrer or change the stirring speed; entry values: 0 to 9; with 0 the stirrer is at a standstill. The change is effected immediately.

– Press this key again to redisplay the current status.

Print

If you press this key twice in quick succession, the stored method and setup parameters are printed out. You can print out the titration, result and setup parameters separately if you press the appropriate key and then press **Print** (see Sections 7.2 and 8).

Stop

When you stop the pretitration, <Run> appears in the display;
when you stop the titration, e.g. "112 ✻ Pre-Titr" appears (see next page).

Esc

If you press this key after an entry/selection before you confirm the parameter with **Enter**, it will not be stored.

Select

You must press this key if you

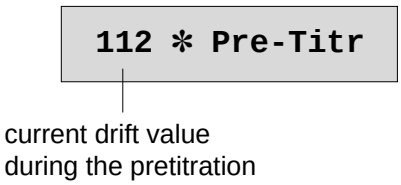
- wish to change the default parameters and values of the method/setup. The selection possibility is marked by an arrow: ↑ (see Section 7.1).
- wish to transfer the weight from an attached balance before or after the titration (see Section 9.1).
- wish to view the potential of the electrode during the pretitration and titration; pressing this key again switches back to the previous display (toggle key).

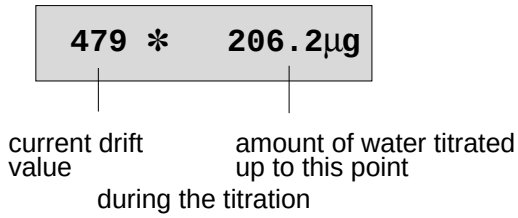
Pot. 201.5 mV	Example with stable drift, this also means that the electrode potential must lie in the vicinity of this value at the end point of a titration.

5.2 The display

The display has space for 16 characters. Depending on the key selection, menu items, parameters or the prompt <Run> for the start of the titration are displayed: The following are shown during the titration:

- the drift in µg/min
- the status ✻ character during pretitration and the titration (sample determination))
  ⇒ character for the start of a titration with the information: "Ready" or "Stable" (see next section)
- the changing, calculated moisture content.





6. The first titration

To determine the moisture content of a sample accurately, parameters are stored in the DL36 which you can change to match your sample. On delivery, the instrument has default parameters stored which you can use to determine the moisture content in µg of many organic substances, e.g. 99% ethanol.

The DL36 is switched on, the display shows **<Run>**.

– Press the **Run** key:

`112 ✻ Pre-Titr`	The pretitration starts; the solution is stirred and titrated until it is free from moisture before you inject the sample.
`5 ⇒ Ready`	An audio signal sounds, this means the solution is anhydrous (ready); however, the drift is not yet stable, its value is ≤500 µg/min. The drift is the amount of water titrated as moisture per minute and recorded. For accurate determinations, you should wait until
`2 ⇒ Stable`	the drift is stable: it now changes by ≤0.1µg per minute. – Prepare the sample: siphon off approx. 0.5 mL 99% ethanol (H_2O content = 0.2%) with a syringe. – Press the **Run** key:
`ID ? ETHANOL`	– Enter the sample identification using the "minus", "period" and numeric keys (max. 10 characters, see Section 8.3). Confirm the entry with **Enter**:
`2 ⇒ Start` `Samp.In.`	"Start" and "Samp.In." (sample injection) appear alternately in the display. – Inject the sample and press the **Run** key:
`978 ✻ 467.2µg`	The DL36 starts the titration and shows the constantly changing drift and calculated amount of moisture.
`3 786.6µg`	When the titration is at an end, an audio signal sounds. The drift and the result are displayed and the result printed out on an attached printer.

- If you wish to determine the second sample immediately, prepare this and press the **Run** key: the sample identification will be requested (see above).
- When you press the **Enter** key, "Stable" (or "Ready") is displayed: the moisture diffusing into the cell will be continuously titrated, in other words the drift value is constantly redetermined (so-called standby titration).
- When you press the **Stop** key, "<Run>" is displayed.

7. The method

A method with default parameters is stored in the DL36. These comprise two groups, the measurement and result parameters, which you call up with the **Titration** and **Result** keys and can adapt to meet the requirements of the individual determinations. You can call up the parameters which support the method using the **Setup** key (see Section 8).

Notes

1. During the titration, the **Titration**, **Result** and **Setup** keys are locked!
2. The stirring speed is not stored as a method parameter and is also not recorded. The stirrer is automatically activated when you start the pretitration (default value = 4). However, you can also activate the stirrer beforehand using the **Stirrer** key. If you switch off the instrument, the stirring speed you last defined remains stored.

7.1 Standard method

Group	Menu	Parameter	Value
Titration		t(stir) [s] ?	0
		t(wait) [s] ?	15
		t(max) [s] ?	0
		Drift stop: rel ↑	
		rel [µg/min] ?	6
Result			
	Calculation ↑	Unit: µg ↑	
		Weight: fix ↑	
		Weight?	5.0000
		Drift comp: off ↑	
		Blank?	0
		Factor	1.00
	Report ↑	Short ↑	
	Recalculation ↑	Weight ?	5.0000
		Drift ?	0.00
		Blank ?	0
		Unit µg ↑	

?: A numeric entry or confirmation of the displayed value is expected.

↑: Menus or parameters have been preset which you can select with the **Select** key. You must then confirm the selection with **Enter**.

7.2 Printing out a method

You can always print out the parameters of a group:

- When you have selected **Titration** and press the **Print** key, the corresponding parameters are printed out.
- When you have selected **Result** and press the **Print** key, the calculation and report parameters are printed out. These parameters are printed out separately when you press the **Print** key while a parameter of the corresponding menu is displayed.

7.3 Modifying a method

You can change the parameters of the groups before and during the pretitration or standby titration.

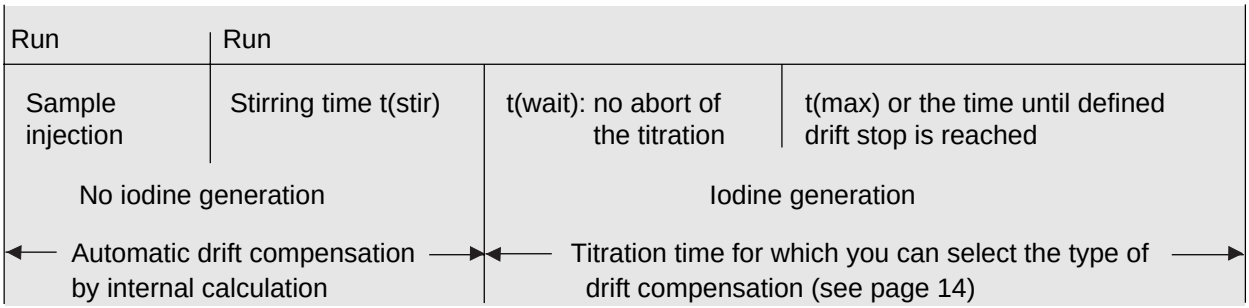
7.3.1 Titration parameters

The parameters are responsible for the control of the titration. You define stirring and titration times as well as drift values which lead to abort of the titration.

Key selection	**Display/Entry** (example)	**Description**
Titration	t(stir) [s]? 0 ..... **10**	Stirring time after the start of the titration: used to ensure thorough mixing or complete release of moisture from the sample; during this time, **no iodine is generated**, in other words no H_2O is titrated.
Enter	t(wait) [s]? 15	Wait time: the titration can not be aborted during this time. A value **<15** is not possible!
Enter	t(max) [s]? 0	t(max) is an abort parameter, the titration is aborted after "t(max) **+** t(wait)" even if the end point has not been reached (t(max) = **0**: abort parameter not defined, see next page).
Enter	Drift stop: rel ↑	The drift stop is the second abort parameter. rel: relative drift stop; the titration is aborted when the drift is less than the "measured drift (before the titration) **+** the defined value" of, e.g. 4 µg/min. abs: absolute drift stop; the titration is aborted when the drift is less than the entered drift value, e.g. 6 µg/min. off: abort parameter not defined. **Notice**: You must define either t(max) **> 0** or a drift stop value, otherwise you receive an error message during the titration. If you define both parameters, the titration will be aborted as soon as one of the values is reached.
Select	Drift stop: abs ↑	
Select	Drift stop: off ↑	
Select	Drift stop: rel ↑	
Enter	rel [µg//min] 6 ..... **4**	Enter, e.g. **4** for the relative drift stop.
Enter		"<Run>", "Ready" or "Stable" is displayed.

Notes

1. Stirring and titration time



The titration time is printed out (Titr. time) if you select "GLP" for the report (see Section 7.3.2).

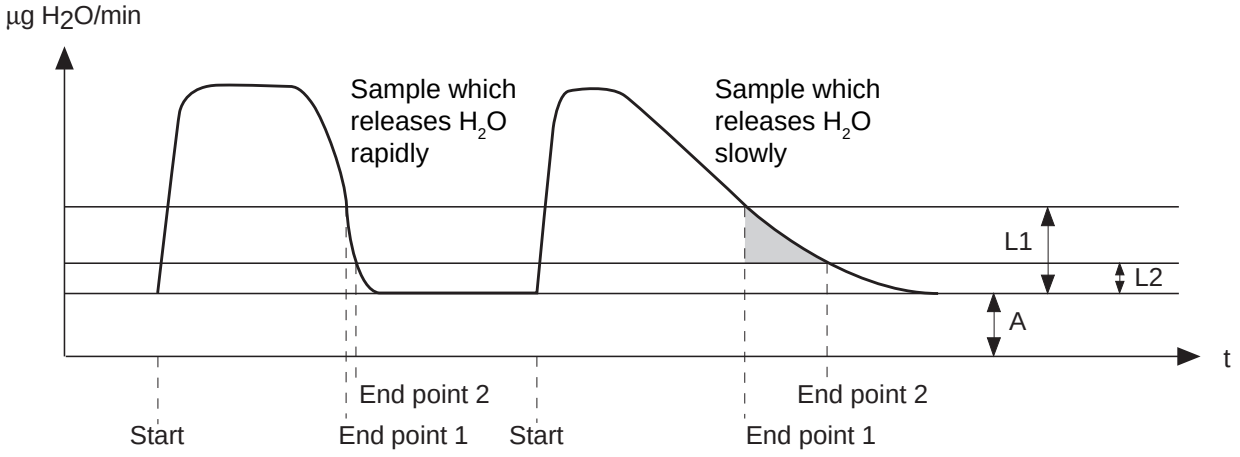
2. Abort parameters t(max) and drift stop

A defined titration time "t(wait) + t(max)" is suitable for titrations in which the end point is reached only very slowly owing to side-reactions.
With t(max) = 0, the titration time depends on the drift stop.

A higher value for the drift stop shortens the duration of the titration, but leads to a larger titration error, above all when the sample releases moisture only slowly.

- With oil samples, for instance, an increase in the drift can be observed. In this case, the end point will not be reached if the drift stop value is too low.
- In the case of titrations with a sluggish approach to the end point as a result of side-reactions, the magnitude of the error can be reduced by a higher drift stop value.

Titration curves with rapid and slow release of moisture with two relative drift stop values:



A: measured drift before the titration
L1: high drift stop value
L2: low drift stop value

End point 1: with high drift stop value
End point 2: with low drift stop value

7.3.2 Result parameters

This parameter group comprises three parts:
1. the calculation parameters (Calculation)
2. the selection of the report (Report)
3. the parameters for the recalculation (Recalculation).

The tables shown follow this subdivision.

1. Calculation parameters

Key selection	Display/Entry (example)	Description
Result	Calculation ↑	
Enter	Unit: µg ↑	Select the result unit for the calculation of the moisture content (the absolute µg content is always recorded). The choice of unit automatically determines the formula used for the calculation (see next page).
Select	Unit: mg ↑	
Select	Unit: ppm ↑	
Select	Unit: % ↑	
Enter	Weight: var ↑	Select the entry of the weight [g]: var: variable weight; inquiry of the weight for every titration. fix: fixed value; the weight for the samples being titrated does not change, the inquiry does not appear again in the titration. If you need only the absolute moisture content without weight value, enter **0** (zero).
Select	Weight: fix ↑	
Enter	Weight? 5.0000 ..... **0.1**	
Enter	Drift comp: off ↑	Select the drift compensation for the titration: off: the drift value calculated before the titration is neither incorporated in the calculation nor recorded. manu: you enter a drift value which is incorporated in the calculation. auto: the drift value calculated before the titration is automatically incorporated in the calculation.
Select	Drift comp: manu ↑	
Select	Drift comp: auto ↑	
Enter	Blank? 0 ..... **2.5**	Enter a blank value [µg], e.g. if you have dissolved a sample in a solvent whose moisture content you have determined beforehand. This blank value is taken into account in the calculation (see next page).
Enter	Factor? 1.0	Enter a factor used to multiply the result, e.g. if you wish to convert the result to an amount of sample which you can not titrate.
Enter	Calculation ↑	To titrate, press **Esc**, to select the report parameters, press **Select**.

Calculation formulae

Formula No.	Result in	Formula
1	µg or mg	$\text{factor} \cdot (T_{value} - \text{drift} \cdot t - \text{blank})$
2	ppm or % (entry of the sample weight)	$\text{factor} \cdot \frac{(T_{value} - \text{drift} \cdot t - \text{blank})}{\text{weight}}$

T_{value}: Result of the titration in µg
drift: Drift value in µg/min
t: Titration time in min (see page 13)
blank: Blank value in µg
weight: Sample weight in g

2. Report parameters

You can choose between two report formats: short-form and GLP format (GLP: Good Laboratory Practice).

Key selection	Display	Description
Result	Calculation ↑	
Select	Report ↑	
Enter	Short ↑	Select either the short-form or GLP format.
Select	GLP ↑	
Enter	Report ↑	To titrate, press **Esc**.

Short-form report

```
Date 96/06/02 13:26
Sample ID ETHANOL
Weight       0.1243 g
Result        219.9 µg
            0.17689 %
```

GLP report

```
Date 96/06/02 13:26
Instr. ID  DL36-82
Sample ID ETHANOL
Weight       0.1243 g
Factor         1.00
Result        219.9 µg
            0.17689 %
Titr.Time  00:00:26
Drift       1.62 µg/min
Blank             0 µg
User ID  KVE
```

On printout:

- Date and time
- Instrument identification
- Sample identification
- Weight
- Factor
- Result
- Titration time
- Drift value
- Blank value
- User identification

3. Parameters for recalculation

If, e.g. you have made a mistake when entering the weight or have selected a different result unit, you can perform recalculations after every sample determination.

Key selection	**Display/Entry** (example)	**Description**
Result	Calculation ↑	
Select	Report ↑	
Select	Recalculation ↑	
Enter	Weight? 0.1243**.....0.1234**	Change the weight if need be.
Enter	Drift? 1.62	Change the drift value if need be.
Enter	Blank? 0	Enter a blank value if need be.
Enter	Unit: µg ↑	Select a different result unit if need be, e.g. **ppm.**
Select	Unit: mg ↑	
Select	Unit: ppm ↑	
Select	Unit: % ↑	
Enter	1756.0	The result is displayed and printed out with the selected report parameters.

8. Setup

Under this key you call up menus and functions which support the method and allow selection of the peripheral devices.

When you press the key, the following appears

Setup[1-8]?

When you enter one of the numbers and confirm with **Enter**, you can define or select the following parameters:

1: Reagent capacity

2: Date & time

3: User identification (User ID)

4: Instrument identification (Instr ID)

5: Peripheral devices (Option)

6: Weight entry (Wt. entry)

7: Entry of the sample identification (ID entry)

8: Initialize default parameters (Memory clear).

Press the **Print** key if you wish to print out the stored data of the menus.

8.1 Checking the reagent capacity

The catholyte and anolyte solutions have a limited capacity for the moisture determination: The capacity limit of

- 100 mL anolyte is reached after the determination of approx. 1000 mg H_2O.
- 5 mL catholyte is reached after the determination of approx. 300 mg H_2O (see specifications of the reagents).

Exhausted reagents result in values of the moisture content which are too low and lead to longer titration times.

The DL36 calculates and sums the amount of moisture in mg which has been determined with anolyte and catholyte and displays these values. You can

- reset these values following reagent change,
- enter limit values for the capacity and
- have the DL36 inform you when these limit values are exceeded.

Key selection	Display/Entry (example)	Description
Setup	Setup [1-8]? **1**	"Reagent capacity" flashes on the display.
Enter	A.Capa. [mg]? 61	Display of the summed electrolytic current for the anolyte – converted to mg H_20. When you change the anolyte, enter **0**.
Enter	C.Capa. [mg]? 61	Display of the summed electrolytic current for the catholyte – converted to mg H_20. When you change the catholyte, enter **0**.
Enter	Alarm set: off ↑	You are not informed when the defined capacity limits are exceeded (see below).
Select	Alarm set: on ↑	You are informed when the defined capacity limits are exceeded (see below). After a titration, the display then shows "A.Capacity Over".
Enter	A.Alarm[mg]? 1000 .. **850**	If necessary, change the default value for the capacity limit of the anolyte.
Enter	C.Alarm[mg]? 300 ... **250**	If necessary, change the default value for the capacity limit of the catholyte.
Enter	Setup [1-8]?	Press **Esc** if you wish to have the current status displayed.

8.2 Entering the date and time

The DL36 has an internal clock. When the instrument is delivered, default values are set for the date and time (95/06/01 and 00:00) from which they will continously update. You should change both values to ensure correct data are reported.

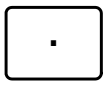
Key selection	Display/Entry (example)	Description
Setup	Setup [1-8]? **2**	"Date ? Time" flashes on the display.
Enter	YY MM DD? 950728 ..... **960718**	Enter year, month and day.
Enter	HH MM? 1051 ..... **1346**	Enter hours and minutes.
Enter	Setup [1-8]?	Press **Esc** if you wish to have the current status displayed.

8.3 Entering the user identification

In this menu you can enter your name; it appears on every GLP report.

You can enter the user and the instrument identification alphanumerically using the "minus", "period" and numeric keys.

When you press this key for the first time, the letter **A** is displayed, for the second time **B** → **C** → ... **Z** → **.** (period) → **–** (minus) → "space" → **A** →...



When you press this key for the first time, a "space" is displayed, for the second time a **–** (minus) → **.** (period) → **Z** → **Y** → ... **A** → **–** "space" →...

You must confirm each letter (character) with **Enter** for it to be accepted.

Key selection	**Display/Entry** (example)	**Description**
Setup	Setup [1-8]? **3**	"User ID" flashes in the display.
Enter	A	Enter, e.g. "A. Aichert": Press the "–" key.
Enter	A_	Press the "**.**" key repeatedly until a period appears.
Enter	A._	Press the "–" key repeatedly until a space appears etc. You can enter maximum 15 characters.
Enter	A. _	
•		
•		

8.4 Instrument identification

On delivery of the DL36, its correct identification is stored (see model plate at the rear); it appears on every GLP report. You can change the identification.

Key selection	**Display/Entry** (example)	**Description**
Setup	Setup [1-8]? **4**	"Instr ID" flashes in the display.
Enter	MMA06408 ..... D	Enter, e.g. "DL36-1A": Press the "**–**" key repeatedly until **D** appears.
Enter	DL	Press the "**–**" key repeatedly until **L** appears.
Enter	DL3	Press the "3" key etc. You can enter maximum 8 characters.
•		
•		

8.5 Selecting peripheral devices

In this menu you select the printer and the balance you have attached to the DL36. For a computer you need to define the corresponding configuration data. The pin assignment of the RS232C interfaces is described in Section 13.2.

1. Printer

You can attach a METTLER TOLEDO GA42 or another commercially available printer. In the latter case, you need to define the configuration data.

Note: If you attach the GA42, you must set its DIP switch 2 to position ON (see GA42 Operating Instructions).

Key selection	Display/Entry	Description
Setup	Setup [1-8]? **5**	"Option" flashes on the display.
Enter	Printer ↑	If "RS232C" or "Balance" is displayed, use **Select** to select "Printer".
Enter	Printer: GA- ↑	Choose between the GA42 and another printer. If you have attached the GA42, confirm with **Enter** (its configuration is preset, see Section 13.2, point 1): "Setup..." is again displayed.
Select	Printer: Other ↑	
Enter	Baudrate: 1200 ↑	Select the baud rate: 300, 600, 1200, 2400, 4800 or 9600.
Enter	Parity: none↑	Select the parity: even, odd or none.
Enter	Data bits: 8 ↑	Select the data bits: 7 or 8.
Enter	Stop bits: 2 ↑	Select the stop bits: 1 or 2.
Enter	Setup [1-8]?	

2. Balance

You can attach a balance from METTLER TOLEDO, A&D, Shimadzu or Sartorius. The configuration is preset for every balance when you select it (see Section 13.2).

Key selection	Display/Entry	Description
Setup	Setup [1-8]? **5**	"Option" flashes in the display.
Enter	Balance ↑	If "RS232C" or "Printer" is displayed, use **Select** to select "Balance".
Enter	Bal: Mettler ↑	Select the name of the company whose balance you have attached.
Enter	Setup [1-8]?	

Notes

1. For all balances you must select the default setting with unidirectional transmission mode "Send Cont.".
2. If you attach a balance using the LC-RS9 cable, you must set positions 7, 3 and 4 (switches left, middle and right).

3. Computer

If you attach a computer, you must select the configuration of the RS232C interface.

Key selection	Display/Entry	Description
Setup	Setup [1-8]? **5**	"Option" flashes on the display.
Enter	RS232C ↑	If "Printer" or "Balance" is displayed, use **Select** to select "RS232C".
Enter	Baudrate: 4800 ↑	Select the baud rate: 9600, 300, 600, 1200, 2400 or 4800.
Enter	Parity: none ↑	Select the parity: even, odd or none.
Enter	Data bits: 8 ↑	Select the data bits: 7 or 8.
Enter	Stop bits: 1 ↑	Select the stop bits: 2 or 1.
Enter	Soft HS: off ↑	Select software handshake: on or off.
Enter	Setup [1-8]?	

You can use the installed computer to transfer data in both directions in a specific format. A detailed description of the communication between the DL36 and a computer can be found in a separate set of instructions.

8.6 Selecting weight entry

You can choose whether you enter the weight before or after the titration.

Key selection	Display/Entry	Description
Setup	Setup [1-8]? **6**	"Wt.entry" flashes in the display.
Enter	Wt.entry:before ↑	You enter the weight before the titration.
Select	Wt.entry:after ↑	You enter the weight after the titration.
Enter	Setup [1-8]?	

8.7 Selecting entry of the sample identification

You can choose whether you enter the sample identification before or after the titration or not at all.

Key selection	Display/Entry	Description
Setup	Setup [1-8]? **7**	"ID entry" flashes on the display.
Enter	ID entry:before ↑	Select between before, after and off. off: no inquiry of the sample identification during the titration, but the identification last entered will be automatically recorded.
Enter	Setup [1-8]?	

8.8 Initializing default parameters

You can delete all entries you have made for the method: The default parameters will then be reentered by the DL36 (see Section 7.1). These include

- the stirring speed (default value = 4)
- the display of the amount of moisture in mg titrated up to this point (default value for catholyte and anolyte = 0)
  the defined limit values for anolyte and catholyte (A = 1000 mg, C = 300 mg)
  the message indicating these limit values have been exceeded does not appear: "Alarm set:off" (see Section 8.1)
- date and time (default values = 95/06/01 and 00:00, see Section 8.2)
- the entry of the weight and the sample number (default setting: entry **before** the titration, see Sections 8.6 and 8.7).

Use the following procedure to set the default parameters:

Key selection	Display/Entry	Description
Setup	Setup [1-8]? **8**	"Memory clr." flashes in the display.
Enter	Memory clr. :off ↑	The initialization is not possible.
Select	Memory clr. :on ↑	The initialization is possible:
Enter	Setup [1-8]?	The confirmation is accompanied by an audio signal. **Notice**: Immediately switch the instrument off, wait five seconds then switch it on again! If you do not do this, undefined values will be entered for all parameters!

After the instrument has been switched on, "SRAM Init." is displayed: the default parameters are initialized.

– Press any key: "DL36 V1.0" appears followed by "<Run>".

9. Sample determinations

9.1 Weight transfer from a balance

You have selected a variable weight as a calculation parameter (see Section 7.3.2) and "before the titration" as the entry for the weight and sample identification (see Sections 8.6 and 8.7).

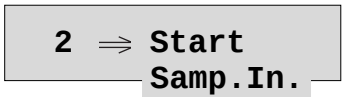
Notice: The mass unit of the balance must be set to **g**!

The anolyte solution is anhydrous, its drift stable (see Section 6).

– Siphon off your sample with a syringe, place the syringe on the balance and tare to **0**.

– Press the **Run** key:

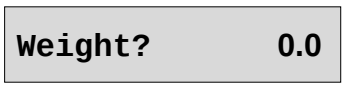
– Enter the sample identification and confirm with **Enter**.



"Start" and "Samp.In." (sample injection) appear alternately in the display.

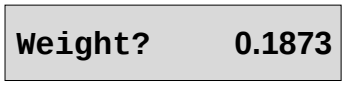
– Inject the sample and replace the empty syringe on the balance.

– Press the **Run** key:



The weight is requested.

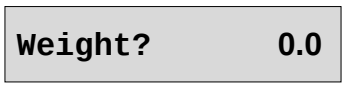
– Press the **Select** key:



The value shown on the balance is transferred.

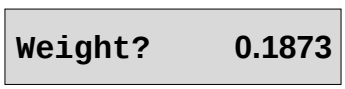
– Press the **Run** key: The titration starts. An audio signal sounds when the titration is at an end; the result is displayed and printed out.

If you have selected "after the titration" as the entry for weight and sample identification, both parameters are requested when the titration is at an end. An audio signal sounds and the following is displayed:



The weight is requested.

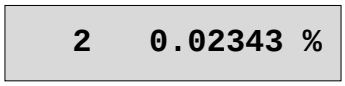
– Press the **Select** key:



The weight shown on the balance is transferred.

– Confirm with **Enter**:

– Enter the sample identification and confirm with **Enter**.



The result is displayed and printed out after the calculation.

9.2 Tips for sample addition

The titration cell of the DL36 is set up for the injection of samples.

1. During sample preparation and sample addition, work as quickly as possible to keep the effect of atmospheric moisture to a minimum. As most samples are stored and transported under standard conditions of temperature and pressure, you should also weigh in and inject under these conditions.
   - You can cool readily volatile samples before sampling (notice: moisture could then condense on the syringe!), viscose samples can be warmed.
   - With hygroscopic liquids, you should never take samples from the surface as the moisture content is highest at this point.
   - Samples in which moisture is present in the form of a dispersion can possibly be treated in an ultrasonic bath before addition.
2. Never remove the Teflon stopper to add solid samples! This would result in an excessive amount of moisture entering the cell.
   - For determination of the moisture content in solids, you can dissolve these in an appropriate solvent or extract them and then inject the solutions. You must determine the moisture content of the corresponding solvent/extraction agent beforehand and enter it as a blank value (see Section 7.3.2).
   - With solids for which no suitable solvent is available for the titration, e.g. polymers, moisture can be driven off only by heating. METTLER TOLEDO offers drying ovens for such situations which lead the resulting water vapor through tubing to the titration cell of the DL36, where it can be determined.
3. Adjust the amount of sample to the expected moisture content to achieve short titration times and allow as many titrations as possible to be performed with the same reagent solutions. The following table provides an overview of the amount to be weighed in.

Moisture content		Sample weight	
50 -	100%	<0,01 g	
10 -	50%	0,02 →	0,01 g
1 -	10%	0,05 →	0,01 g
0,1 -	1%	0,1 →	0,01 g
0,01 -	0,1%	1,0 →	0,1 g
0,001 -	0,01%	5 →	1,0 g
0,0001 -	0,001%	10 →	5 g

not suitable for coulometry

9.3 Tips for accurate determinations

1. We advise always using the DL36 in the operating mode "Pretitration" (standby titration).
2. Always wait for a stable drift before you inject the sample. This allows determination of an exact value for the drift change per minute, which is incorporated in the calculation of the result.
3. Titrate the cell overnight when you determine samples whose absolute moisture content is less than 50 µg (standby titration).
4. With a low moisture content, rinse the syringe with the sample before you perform the first titration.
5. Check the DL36 by measurements with a reference substance, e.g. Hydranal® check solution from Riedel-de Haën.
6. Change the reagents when
   - the anolyte solution reaches a volume of 150 mL (second mark on the titration cell),
   - the capacity of the catholyte or anolyte is exhausted (see Section 11.6),
   - the drift is too high (>10 µg/min).
7. Always titrate a sample series with the same stirring speed.

9.4 Applications

We have developed applications for volumetric and coulometric titrations which can be used to determine moisture in solvents, petroleum products, plastics, foods and cosmetics. The applications include

- instructions for sample preparation
- advice for substances which cause side reactions in the titration
- literature references.

They are listed in Section 12.2.

10. Error messages and malfunctions

10.1 Error messages of the DL36

Error message	Cause	Measures
A.Capacity Over!	The summed electrolytic current (converted to mg H_2O) of the anolyte is greater than its capacity	• Change anolyte • Set capacity of the anolyte to **0** (see Section 8.1)
C.Capacity Over!	The summed electrolytic current (converted to mg H_2O) of the catholyte is greater than its capacity	• Change catholyte • Set capacity of the catholyte to **0** (see Section 8.1)
Current error!	There is no current flowing between anode and cathode	• Check anolyte and catholyte • Check supply leads of cathode and anode
Elect open!	The detection electrode is not working properly (break)	Plug in electrode correctly
Elect short!	The detection electrode is not working properly (short circuit)	Rectify short circuit (align platinum pins correctly)
Meas. Over!	Measurement range exceeded. The amount of moisture in this sample is greater than 100 mg H_2O	Change catholyte, inject smaller amount of sample
Over Titr.!	Overtitrated: the anolyte contains an excess of iodine	• Clean anode • Protect instrument against direct sunlight
Para set miss!	Neither of the two abort parameters t(max) and drift stop is defined	Define either t(max), the drift stop or both parameters
Pre Amp Err-XX	Error in the preamplifier	Contact METTLER TOLEDO Service

10.2 Other malfunctions

Fault	Possible causes	Measures
Instrument can not be switched on	No line voltage Power cable not connected Fuses faulty	Check power cable, on/off switch, voltage selector and fuses
Malfunctions of the stirrer	Stirrer speed set to **0**	Check stirring speed (stirrer key and size of the magnetic stirrer (35 mm)

Fault	Possible causes	Measures
Drift too high	Leaks in the system	Change septum and drying agent, (apply very thin coating of grease to glass joints)
	Capacity limits of the anolyte/catholyte reached or "wrong" reagent used	Change reagents
	Membrane wetted with moisture or cracked	Clean inner burette or replace
	Anode touching the membrane	Insert anode adjustment element between membrane and anode and set a distance between 1.5 mm and 2 mm
	Detection electrode contaminated or faulty	Clean/replace detection electrode
Reagent consumption too high	Reagents exposed to direct sunlight	Protect instrument against direct sunlight
	Detection electrode contaminated or faulty	Clean/replace detection electrode
	Capacity limits of the anolyte/catholyte reached	Change reagents
	Deposits on the anode surface	Clean inner burette
	Anode touching the membrane	Insert anode adjustment element between membrane and anode and set a distance between 1.5 mm and 2 mm
End point not reached or reached too late	Drift stop set to "off"	Set "drift stop" to "on" and enter value
	Drift stop value too low	Enter a larger drift stop value
	Side reactions	Use a different KF reagent (e.g. for ketones)
	Capacity limits of the anolyte/catholyte reached	Change reagents
	Detection electrode contaminated or faulty	Clean/replace detection electrode
Poor reproducibility of the results	Moisture content of the sample too high/low	Adjust amount of sample (see Section 9.2)
	Drift too high	See "Drift too high".
	Reagent consumption too high	See "Reagent consumption too high"
	Drift stop value too high	Enter lower drift stop value
	Capacity limits of the anolyte/catholyte reached	Change reagents

11. Maintenance and servicing

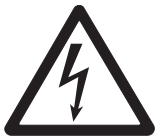
11.1 Charging internal battery

A battery is responsible for the internal clock. Contact the METTLER TOLEDO Service when it is discharged, i.e. when the date and time are no longer correctly displayed.

11.2 Line voltage, line fuses

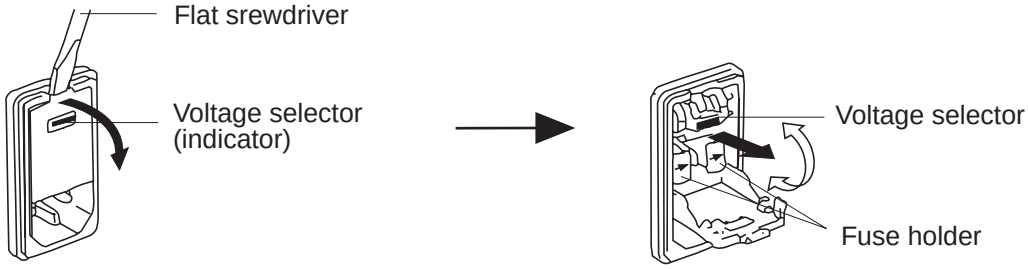
The DL36 can operate in different voltage ranges. The voltage selector is used to select the range.

Voltage range/supply	Setting on voltage selector	Fuses
100 V, 50/60 Hz 120 V, 50/60 Hz	100 VAC 120 VAC	T3,15L250V
220 V, 50/60 Hz 230/240 V, 50/60 Hz	220 VAC 230/240 VAC	T1,6L250V



Changing voltage/fuses

– Switch the instrument off and disconnect the power cable!
– Open the cover above the power cable socket using a flat screwdriver.



– Take out the drum of the voltage selector and reinsert horizontally with the desired voltage.
– Remove both fuse holders using a screwdriver and insert the appropriate fuses (see above).
– Reinsert the fuse holders (note direction of arrow) and replace the cover.

The same procedure applies to the replacement of blown fuses.

11.3 Greasing the glass joints

– Turn the stopper/electrode/drying tube of the titration cell about once a week to check their contact and to prevent them from sticking.
– Apply a very thin coating of grease if the contact (tightness) is no longer assured.

Notice: If an execessive amount of grease is applied, it could enter the titration cell and increase the drift owing to the moisture it contains!

11.4 Changing the septum

– Change the septum of the injection port at frequent intervals. As old septums can easily break, air can enter the titration cell and increase the drift.

11.5 Changing the silica gel

You should change the silica gel as soon as it starts turning pink.
– Unscrew the cover.
– Change the silica gel and screw the cover back on.

11.6 Changing the reagents

WARNING Switch the DL36 off and place it in a fume hood! Never inhale the vapors of Karl Fischer reagents and avoid skin contact! The reagents could hazard your health.

1. Catholyte

You should change the catholyte when
- the capacity limit is reached (see Section 8.1),
- the drift is too high (>10 µg/min) or the status "Stable" is no longer reached,
- the membrane is contaminated.

Changing the catholyte at regular intervals avoids high drift values and measurement errors.

– Siphon off the catholyte using the polyethylene bottle.
– Add 5 mL catholyte using a syringe (up to lower mark on the titration cell).

Notice: After the change, select **Setup 1** and enter **0** for "C.Capacity". This sets the value of the summed electric current (converted to mg H_2O) of the catholyte to zero (see Section 8.1).

2. Anolyte

You should change the anolyte when

- the capacity limit is reached (see Section 8.1),
- the drift is too high (>10 µg/min) or the status "Stable" is no longer reached,
- its volume in the titration cell has reached 150 mL (upper marking on the titration cell); if the anolyte is diluted too highly, the sensitivity is lowered and the titrations take longer.

– Siphon off the anolyte using the polyethylene bottle.
– Add 100 mL anolyte to the titration cell (up to the lower marking on the titration cell).

Notice: After the change, select **Setup 1** and enter **0** for "A.Capacity". This sets the value of the summed electric current (converted to mg H_2O) of the anolyte to zero (see Section 8.1).

11.7 Cleaning the detection electrode

– If the platinum pins of the electrode are contaminated, first clean with concentrated nitric acid or carbon tetrachloride and then with methanol.

11.8 Cleaning and drying the inner burette

WARNING Place the DL36 in a fume hood! Never inhale the vapors of Karl Fischer reagents and avoid skin contact! The reagents could damage your health.

1. Cleaning with methanol

– Switch off the DL36.
– Remove the inner burette from the titration cell and allow the catholyte to flow out.
– Degrease the joint surfaces with methanol.
– Rinse out the cathode of the inner burette two or three times with methanol.
– Add approx. 10 mL methanol and place in a beaker.
– Fill the beaker with methanol until the methanol level in the cathode is reached.
– Allow to stand for around 30 minutes, then empty the inner burette and dry it (see point 3).

2. Cleaning with chromic acid (if foreign matter has collected on the membrane or the platinum surface).

– Perform the first 3 steps as described under point 1.
– Add approx. 10 mL chromic acid and place in a beaker.
– Fill the beaker with chromic acid until the level in the cathode is reached.
– Allow to stand overnight.
– Empty the inner burette and wash the cathode five or six times with pure water until the yellow color disappears (rinse chromic acid from the membrane).
– Rinse with methanol and dry.

Note: Chromic acid cleaning solution: dissolve approx 1.5 g potassium dichromate in 100 mL concentrated sulfuric acid.

3. Drying

– Place the inner burette in a vacuum drying oven for at least two hours (temperature no higher than 50 °C).

If no vacuum drying oven is available, you can use a hair dryer (blow warm air over the membrane of the inner burette until it is dry) or a desiccator.

11.9 Measures for long-term storage

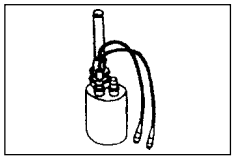
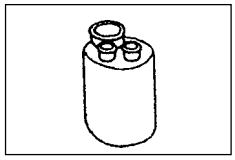
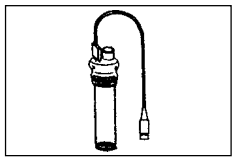
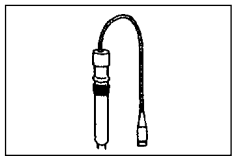
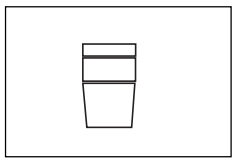
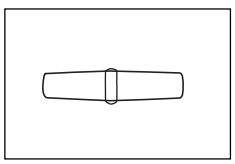
We recommend cleaning the titration cell, disassembling it, drying the parts and then storing them in a desiccator.

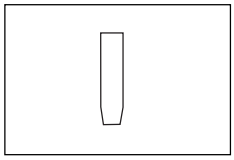
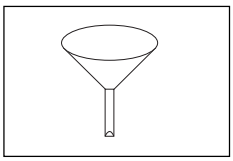
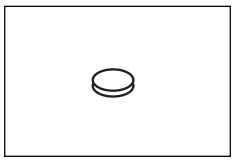
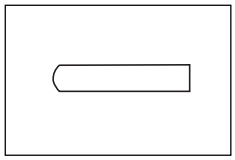
12. Standard and optional equipment

You can reorder every part with an order number; the numbers in brackets refer to the quantity supplied with the DL36.

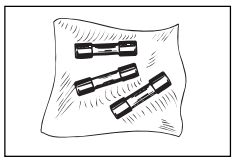
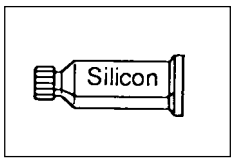
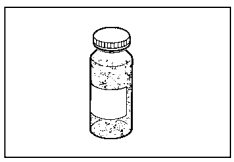
12.1 Standard equipment

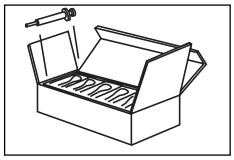
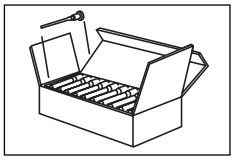
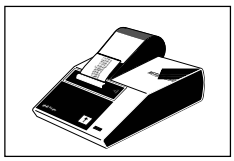
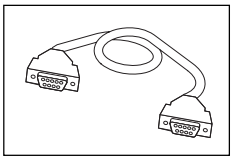
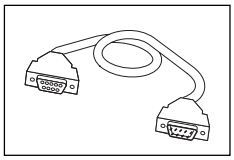
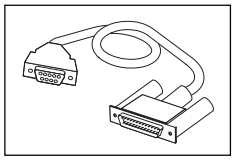
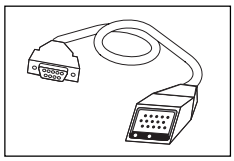
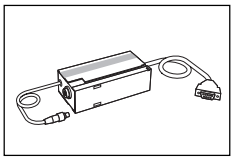
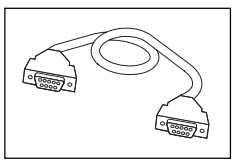
	Order No.
DL36 KF Coulometer	
1 set Operating Instructions	in accordance with your order
1 memo card	in accordance with your order
1 power cable	in accordance with your order
1 set fuses (3)	in accordance with your order
1 jar silicon grease	–

	Titration cell, complete **comprising** Silicon stopper	51107410 –
	Titration cell	51107411
	Inner burette	51107412
	Double-pin platinum electrode	51107413
	Teflon stopper with septum (injection port)	105017
	Magnetic stirrer (1) length 35 mm	105053

			Order No.
	Drying tube (1) • filled with silica gel		105016
	Suction bottle made of polyethylene (1)		105123
	Plastic funnel (1)		105124
	Septum (10), Ø: 12/2 mm	set of 10	105074
	Anode adjustment element (1)		105011

12.2 Optional equipment

	Fuses T3,15L250V T1,6L250V	 set of 3 set of 3	 54286 18560
	Silicon grease		71300
	Silica gel (1 kg)		105080

		Order No.
	Syringe 1 mL set of 100 Syringe 10 mL set of 100	71492 71482
	Injection needle set of 12 80 mm x 1.2 mm	71483
	METTLER TOLEDO printer	GA42
	Cable for GA42 Printer	51190362
	Cable for DPU-201GS printer from Seiko Instruments	51106171
	Cable for InkJet printers from Canon, HP and EPSON (D-submin. 9-pin, female, D-submin. 25-pin, male)	51190363
	Cable for AT balance	229029
	LC-RS9 cable for AG, AB, PB, and PR balances	229065
	Computer cable (RS232C) DTE (D-submin. 9-pin, female, D-submin. 9-pin, female)	51190362

		Order No.
Operating Instructions	German	51709541
	English	51709542
	French	51709543
Memo card	German	51709585
	English	51709586
	French	51709587
METTLER TOLEDO brochures		
Karl Fischer applications for	Foods, beverages, cosmetics	724478
	Chemicals, solvents, petroleum products, plastics	724354
	Karl Fischer The method at a glance	724573

13. Technical data

13.1 DL36

Measurement method	coulometric titration following Karl Fischer
Control system	timing control by the microprocessor with constant current pulses
End point detection	by alternating current polarization
Status display for a titration	Ready: Titration possible for rough results Stable: Titration possible for exact results
End point display	by audio signal
Stirring	with magnetic stirrer, length 35 mm
Capacity of the titration cell	maximum 150 mL
Measurement range	10 µg to 100 mg H_2O content of a sample
Resolution	0.1 µg
Repeatability	the relative standard deviation of the titration of 1 mg H_2O in methanol is less than 0.3 %.
Measurement time	90 - 120 seconds for the titration of 1 mg H_2O in methanol
Drift	automatic compensation for the result calculation selectable
Display of the amount of moisture	from 0.1 µg to 999999 µg (after the titration)
Entry range for • stirring time t(stir) • wait time t(wait) • t(max) • drift stop	 0 - 999 seconds 15 - 999 seconds 0 - 9999 seconds 0 - 999 µg/min
Calculation function	Calculation of the moisture content • in µg or mg • in ppm or % with known weight

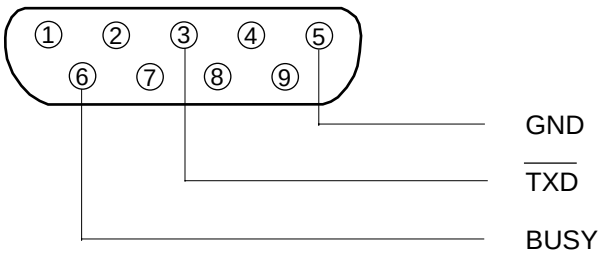
Housing	Polyester
Titration cell	Pyrex®
Display	LCD, 16 characters
Dimensions	width: 260 mm, depth: 395 mm, height: 270 cm
Weight	approx. 6.4 kg
Power supply	100/120/220/230/240 VAC ±10% 50/60 Hz 50 VA
Fuses	T1,6L250V; T3,15L250V
Ambient conditions	• use only indoors • height up to 2000 m • ambient temperature: +5 to +35 °C • maximum relative atmospheric humidity 80% for temperatures up to 31 °C, linearly decreasing down to 50% relative humidity at 40 °C • overvoltage category: ll • pollution degree: 2

13.2 Interfaces of the DL36

1. Printer interface

RS232C interface for the attachment of a GA42 Printer or for various commercial printers

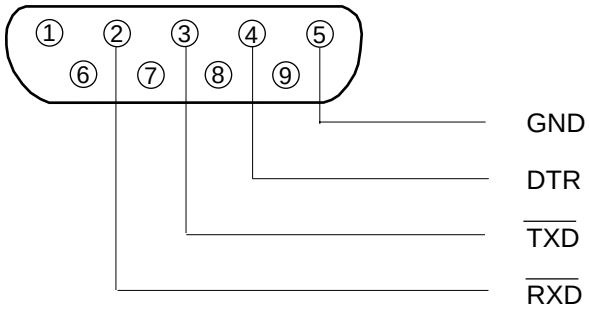
Pin assignment



2. Balance interface

RS232C interface for the attachment of a balance with unidirectional transmission mode "Send Cont."

Pin assignment



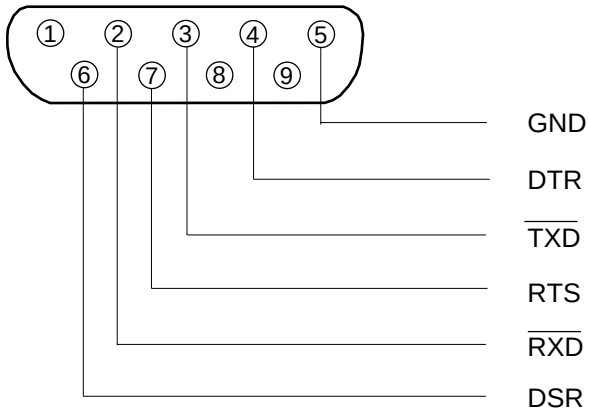
Preset configuration on attachment of one of the possible balances from:

Selection / Default	Mettler	Shimadzu	A & D	Sartorius
Baud rate	2400	1200	2400	1200
Parity	even	none	even	none
Data bits	7	8	7	8
Stop bits	1	1	1	1

3. Computer interface

RS232C interface for the attachment of a computer or terminal

Pin assignment



14. Index

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Thank you.

Printed on 100% chlorine-free paper, for the sake of our environment.

P51709542

Subject to technical changes and to the availability of the accessories supplied with the instruments.

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